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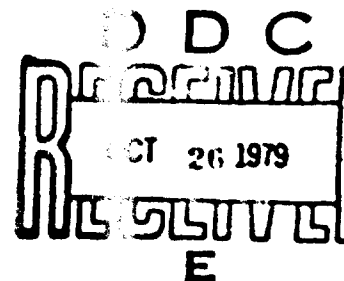
OVERVIEWS OF HNS PRODUCTION / PROPERTIES / APPLICATIONS

BY E. EUGENE KILMER

RESEARCH AND TECHNOLOGY DEPARTMENT

3 JULY 1979

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21. ABSTRACT (Continue on reverse side if necessary and identify by block number) Hexanitrostilbene (HNS) has been used within the U.S. government/industrial complex since the early 1960's. Chemical process variables leave much to be desired in terms of product purity and control of particle geometry. The explosive HNS-I, from the Shipp process, is normally fine, flat platelets which have been found to contain upwards to 6% hexanitrobiphenyl (HNBIB) or dipicrylethane (DPE) as an impurity. Attempts to remove this impurity by multiple washings has resulted in recrystallized material of a new geometry and		

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consequently larger particle size. The large particle size is undesirable in many applications. The explosive HNS-II, first recrystallized by the Taylor-Oesterling process, is usually accomplished by extraction by dual-organic solvents. Most recently, HNS has been recrystallized from nitric acid. The thermal stability of the HNS recrystallized from nitric acid appears to be different from the HNS recrystallized from organic solvents as demonstrated in the low core load detonating cords. The chemical/explosive properties will be discussed.

HNS has found many applications throughout the aerospace industry in explosive components for high speed aircraft and spacecraft and has been incorporated into a PBX seismic charge. The properties of this PBX will be discussed.

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This report covers a review conducted in part for the NASA Lyndon B. Johnson Space Center, Houston, Texas under NSWC Task R12ZB. Hexanitrostilbene (HNS), a thermally stable explosive, has been qualified for applications in the NASA complex and in many Navy applications. The explosive is synthesized by several vendors in the United States using the Shipp process. Variable purities have been found with the major impurity being hexanitrobibenzyl (HNB) or dipicrylethane (DPE). The HNS is used in detonating cords and large explosive charges. The author wishes to acknowledge the detonation velocity work done by Mr. Charles Goode, the chemical analyses by Ms. Eleonore Kayser, and the scanning electron photomicroscopy by Dr. Harriner Norr.

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CONTENTS

	Page
Introduction.....	5
HNS Synthesis/Particle Geometry.....	5
Availability of HNS Explosive.....	7
Application of Hexanitrostilbene.....	9
Summary.....	9
References.....	11

ILLUSTRATIONS

Figure	Page
1 HPLC Trace of a Synthetic Mixture of Potential HNS Impurities (NSWC).....	13
2 SMDC End Booster... ..	13
3 Crystal Growth HNS-I.....	14
4 Detonation Transfer Arrangement McDonnell Douglas Cooperation	15
5 Hexanitrostilbene-Fragment Initiation Sensitivity at a 50% Fire Response with Steel Barrier Thickness vs Explosive Surface Area at the 0"500 Air Gap.....	16
6 Hexanitrostilbene-Fragment Initiation Sensitivity at a 50% Fire Response with Steel Barrier Thickness vs Explosive Surface Area at the 0"100 Air Gap.....	17

TABLES

Table		Page
1	Quantitative HNS Data.....	18
2	Availability-Hexanitrostilbene (HNS).....	18
3	Detonation Velocity/Chemical Analysis after Elevated Temperature Storage @425°F HNS-II Silver MDC.....	19
4	Explosive Technology SMDC Summary (Major Programs Only).....	20
5	Physical and Explosive Properties of Hexanitrostilbene, HNS/ TEFLON Compared to RDX.....	21

Introduction

Hexanitrostilbene (HNS) has been produced in the United States by the Shipp process^{1,2} since the early 1960's. This one step process, from TNT and commercial bleach, was the only synthetic route to HNS for many years. Earlier work by Reich,³ reporting on findings on the preparation of hexanitrostilbene, shows a melting point of 211°C, but it is believed that this material was probably hexanitrobibenzyl (HNBiB) instead of hexanitrostilbene as claimed. Recent literature shows effort by Stull^{4,5} and Clink⁶ to improve the quality of the HNS, the efficiency of the Shipp process and alternate routes of synthesis.

The Hungarian patent by Tompalthy⁷ et al, suggests a route of synthesis for HNS by using an oxidation catalyst; a metal complex of cobalt or copper. A duplication of this work has met with little or no success in the United States. The British have patented a process as an improvement over the Shipp process by reacting TNT in the presence of ammonia or an amine.⁸

HNS Synthesis/Particle Geometry

It is inherent in the synthesis of HNS that many side reactions take place forming a number of impurities which are caught up in the HNS crystal on precipitation from the red tar fraction. Kayser⁹ at the Naval Surface Weapons Center (NAVSWC) has identified at least twelve by-products of the reaction by using a combination of thin layer chromatography (TLC) and nuclear magnetic resonance spectroscopy (NMR).

In addition, Schaffer¹⁰ has developed a method to analyze HNS samples using high pressure liquid chromatography (HPLC). The impurity appearing in the largest percentage is the hexanitrobibenzyl. A graphical representation of the appearance of these materials as they occur on the HPLC reverse-phase column⁹ is pointed out in Figure 1. This synthetic mixture contains the basic impurities found in HNS although all of these do not appear in every lot of HNS that is produced from the methods indicated above. Each vendor will usually have a range of percentages of various impurities depending on the method and the process control. A tabulation of the results of a number of chemical assays from HNS produced by several vendors can be seen in Table 1. These values should be considered to be typical and not necessarily limited to the single percentages of compounds indicated on the table. For example, HNS-I, prepared by vendor C, will assay

at 95.9% HNS depending on the range of assay for the HNBIB. Empty columns are indicative of purity from that particular compound. Since the source column letters do not duplicate identical vendors for HNS-I and HNS-II, the % TNT column indicates three vendors had some percentage of the original, unreacted starting material appearing in the final product. From a chemical purity viewpoint, these impurities are undesirable but it is probably impractical to "clean-up" the product unless it is justifiable. This brings up the question of how is the crystal geometry effected by any "clean-up" or whether one should be concerned with any change in the geometry of the HNS crystal at all? Unfortunately, the shape of the HNS crystal has a significant effect on the explosive response to fragment initiation. In addition, in small diameter explosive train hardware, this can also be critical to the reliability of performance if there is any significant change in the particle geometry of the acceptor explosive. To get this into perspective, a requirement for a new type explosive component was developed in the early 1960's to meet the needs of the F-111 Aircraft Program. This explosive component had both capabilities of being a donor and/or an acceptor. Peculiar to the design was the fact that after much expenditure of time and money, the design could be faulted only when the recrystallized, large particle size HNS was used throughout the component. The thermal requirements were well below any temperature which would cause problems with the relatively impure HNS which crystallized out of the red tar fraction of the organic synthesis. It was discovered at the outset of the testing of the explosive component, that only the thin platelets of HNS-I would support a reliable detonation transfer. The explosive component in question was the shielded mild detonating cord (SMDC)^{11,12} and is shown in Figure 2. The end coupler charge of HNS is the key to the reliable performance of the component when the end coupler is the donor. The base charge section is the key to reliable detonation transfer when the end coupler is the acceptor. Several papers have been written describing earlier work^{13,14} on the development of the end coupler for the SMDC line. The point is the particle shape/size of the HNS significantly influences the performance of the SMDC explosive component. A photomicrograph of the HNS-I as it was first synthesized is shown in Figure 3. The HNS-I sample, ID 714, is typical of the particle size and shape of the explosives used in the end coupler, and is typical of the HNS from the Shipp process. On the same illustration, a photomicrograph is shown of HNS-I (ID 1987) explosive produced by the Shipp process but allowed to stand in large holding tanks for many hours awaiting the next step of the process. ID 1987 is typical of HNS-I which has been allowed to grow under conditions of large volume recrystallization.

However, in view of the crystal growth, particle size, bulk density charge, change in explosive sensitivity, etc., HNS-I is a misnomer for this lot of HNS. This type of HNS explosive is considered to be an HNS-I/HNS-II hybrid for further reference. For example, a similar lot of HNS-I was tested in the laboratory for fragment initiation sensitivity. It was fabricated into the base charge section of the SMDC component. This unit was used as an acceptor and tested with a standard SMDC tip as the donor. In this special case, a special thickness of steel barrier was specified over a range of 0.003 to 0.025 to be implemented by using steel shims of variable thickness to be substituted for the cup bottom of the acceptor. The donor and acceptor were set up at 0.500 and 0.100 air gap between them for the two

tests as shown in Figure 4. The shim thicknesses were varied for the tests, and the results of these test firings, with a limited number of shots at various barrier (cup) thicknesses, are shown in Figures 5 and 6. The results are plotted as the 50% fire response from the SMDC donors' fragment stimulus. The results from tests in the end-to-end initiation configuration as shown in Figure 5 are from the typical arrangement of this ordnance in actual application; except for the penalized air gap. Since this has been the accepted design for at least 15 years, reduced thickness barriers are as expected, even for the decreased sensitivity of the HNS hybrid material. The results in Figure 6 indicate HNS-I is an acceptable material in the cup of the SMDC tip even at 0" air gap in a side-to-end initiation configuration. The results are consistent with the increase in explosive surface area (reduced particle size), the donor remaining constant, the steel barrier thickness can be increased at the acceptor without reducing the reliability of initiation of the acceptor. The question of integrity of the SMDC system is centered about the plot of data in Figure 6 where a complete reversal in the performance of the acceptor is shown as the surface area decreases and the barrier thickness drops below five mils thickness with the HNS-II loading. The side-to-end initiation of an acceptor is one of the most undesirable methods of initiation at best but to combine this with more insensitive explosives in the acceptor SMDC line is asking for reduced performance in the form of a failure to initiate at design barrier thickness parameter (cup bottom). It points out that HNS-II is not acceptable in the end cup and is an indication that any hybrid HNS explosive could approach this area of design margin if the surface area became small enough. Even in view of the small number of tests, the results are conclusive and the standard deviation of ± 2 mils would not be expected to change with an increase in the sample size. After reviewing over 50 separate lots of HNS-I from industry in the United States and three lots from England, there have been only two lots of HNS-I from the United States and none from England which showed this HNS-I hybrid structure. Therefore, only 4% of the lots produced in the United States have been in holding tanks for an abnormally longer time period than with the other HNS processes. The washing procedures for HNS-I should be carefully controlled but the probability of approaching the "worst-case" situation in crystal growth is small. A change in the margin of performance in SMDC end boosters has been defined but it is not expected to effect the ultimate performance of the explosive hardware.

This discussion has been pointed entirely toward a specific piece of ordnance: the SMDC explosive component. The choice of whether the designer should use HNS-I or HNS-II depends on various constraints of environment and mechanical design on the final design which is on a per case basis. Aside from component usage, a recent demand for HNS as a nucleating agent in the control of the recrystallization of TNT¹⁵ during casting has been cited. The literature does not define which HNS to use in this process. Unless there is some technical reason influencing the behavior of the TNT by using HNS-I as opposed to HNS-II, then the more economical route would be to use the HNS-I.

Availability of HNS Explosive

The availability of both HNS-I and HNS-II has been increasing over the

last five years. Teledyne McCormick Selph, United Technologies, Mason and Hanger and Ensign Bickford have been added to the list as shown in Table 2. Note that some companies have chosen to make only HNS-I or HNS-II and some have chosen to make both types of materials. The British have produced HNS-I by their modified Shipp process and are using it to improve the crystallization or nucleation of the TNT melt in shell explosive loading facilities.

All of the companies currently producing HNS-I and HNS-II have been investigated. Historical samples have been collected from the early American Cyanamid productions and from the latest products available by all vendors. Inherent in the HNS synthesis, is the production of hexanitro-bibenzyl (HNBiB) and small amounts of the impurities. The production of the HNBiB depends primarily on the temperature control of the reaction mixture and on the temperature at which the HNS reaction is carried out. It is rather difficult to remove except through many washings as has been pointed out earlier. In Table 1 the column heading Σ "P.P." is defined as unknown material/materials which requires more study to analyze. Recrystallization of HNS removes the majority of these impurities but definitely changes the crystal geometry.

The recrystallization of HNS-I has been studied by Taylor,¹⁶ O'Keefe,¹⁷ Sandoval,^{18,19} Quinlin,²⁰ and Syrop²¹ using organic solvents. Taylor studied the recrystallization of HNS to improve the bulk density of the material for loading into mild detonating cord (MDC), and flexible linear shaped charge (FLSC). He found that acetonitrile/toluene was effective for HNS as a double solvent system in the continuous extraction-recrystallization apparatus. Syrop also found continuous extraction-recrystallization to be effective with acetonitrile/xylene according to his patent. The flow properties of the recrystallized HNS is satisfactory for loading into MDC and FLSC. This particular application of HNS-II is the largest of all the uses of HNS-II. It has been produced from lot sizes of a few kilos to 45 Kg lots in the United States. Laboratory size glassware and stainless steel kettles are being used to recrystallize the HNS now being produced in the United States. All vendors have proprietary rights in the processing of the material but a careful review of the end product reveals a crystal geometry characteristic of the solvent/solvents used in the process. In the manufacture of HNS-II, the demand or the requirement for a particular particle size/geometry is not as critical in the performance of the end product hardware as was HNS-I. Both HNS-I and HNS-II can be loaded into cords but the HNS-II has the more desirable flow characteristics.

Two vendors, Chemtronics and Teledyne McCormick Selph supply HNS-II processed from nitric acid solvent. This method of recrystallization of HNS was not accomplished until the early 1970's. The processes are still proprietary to both companies. The crystal geometry and size appear to be controllable but is inherently large. The flow properties of the HNS and the more economical processing of the explosive makes it more attractive to the user but not without a serious drawback. The problem with this material when loaded into detonating cords is that the thermal stability of the explosive is penalized as is shown in Table 3. This is substantiated by poor performance demonstrated by the MDC when compared to the performance of the

organic solvent recrystallized material tested under the same conditions.^{22,23} The work published by Gould²⁴ at the Sandia Laboratories points out the limiting factor in the thermal stability of HNS produced currently in the presence of dipicrylethane (DPE) or hexanitrobibenzyl.

The availability of HNS within a short time frame is variable depending on the demand and the availability of solvents, however most of the vendors will furnish the explosive in 45 Kg lots.

Application of Hexanitrostilbene

HNS has found many applications throughout the aerospace industry in explosive components for high-speed aircraft and spacecraft. It has also been incorporated into a plastic bonded explosive charge (PBX). Special components such as the SNDC line, as shown in Table 4, is the largest user of HNS. As has been pointed out, HNS-I must be used in the end coupler/end tip arrangement. Many thousands of these units have been fabricated and tested to a high demonstrated reliability.

The largest explosive charges made with HNS were fabricated for the APOLLO 17 Lunar Seismic Profiling Experiment.²⁵

In these charges, HNS-II was blended with Teflon-7C (E. I. duPont deNemours Co. Trademark) isostatically pressed, and machined to the proper shape for the application by Misener.²⁶ A better understanding of the thermal properties of the explosive and inert binder has been published by Elban,²⁷ who developed values of thermal diffusivity and thermal conductivity for the explosive blend and the inert simulant for the charge. Additional properties are shown in Table 5. Montesi²⁸ studied the explosive sensitivity of HNS/Teflon 90/10 by a series of verification tests. The probabilities of detonation transfers between the in-line explosive components were determined by the VARICOMP test²⁹ method and exceeded 0.9999 at 95% confidence for all interfaces in the explosive train. The explosive charges were subjected to vigorous vibration testing and were accepted for flight environment. The explosive charges fabricated for this mission range from booster size (60 gm) to main charge size (2.7 Kg). It demonstrates the usefulness of the explosive blend and future possibilities in explosive train design. At the Lawrence Livermore Laboratory (LLL), Golopol³⁰ blends 95 wt% HNS-I and 5 wt% Kel-F 800 to form an acceptable explosive composition for use as an explosive booster. Several papers have been written on the detonation properties of HNS where Hanea³¹ measured the unreacted Hugoniot of HNS and Lee³² developed an equation of state for the detonation products of HNS at various charge densities.

Summary

The Shipp process is probably the most used and most economical synthesis of HNS-I at the present time in the United States. The Taylor-Oesterling recrystallization process for HNS-I to form HNS-II, yields material which has better thermal stability than the material recrystallized from nitric acid. All HNS produced in the United States by qualified vendors appears to retain the thermal qualities of the basic explosive material.

There is however, a considerable percentage of impurities. Hexanitrostilbene recrystallized from nitric acid, should not be used in detonating cords. Manufacturers of HNS-I should be aware of crystal growth brought about by retaining the explosive in holding tanks for long periods of time.

HNS-I or HNS-II may be combined with a proper binder and pressed into an acceptable explosive charge with good mechanical and thermal properties.

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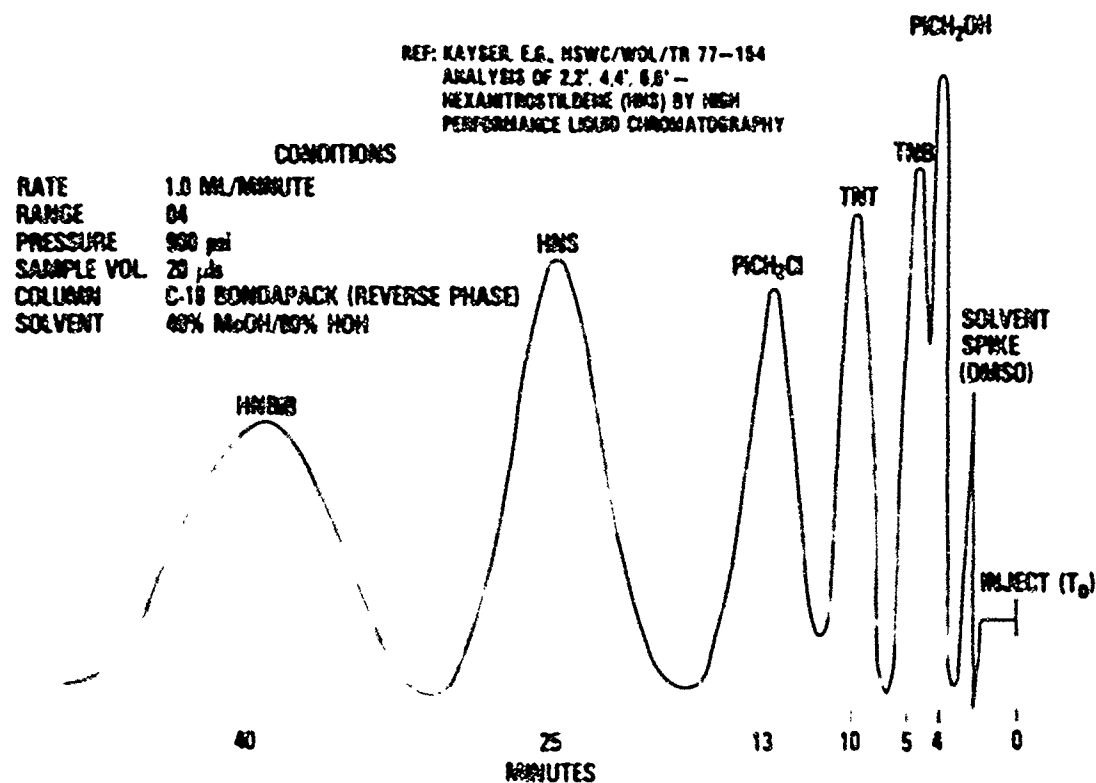


FIGURE 1 HPLC TRACE OF A SYNTHETIC MIXTURE OF POTENTIAL HNS IMPURITIES (NSWC)

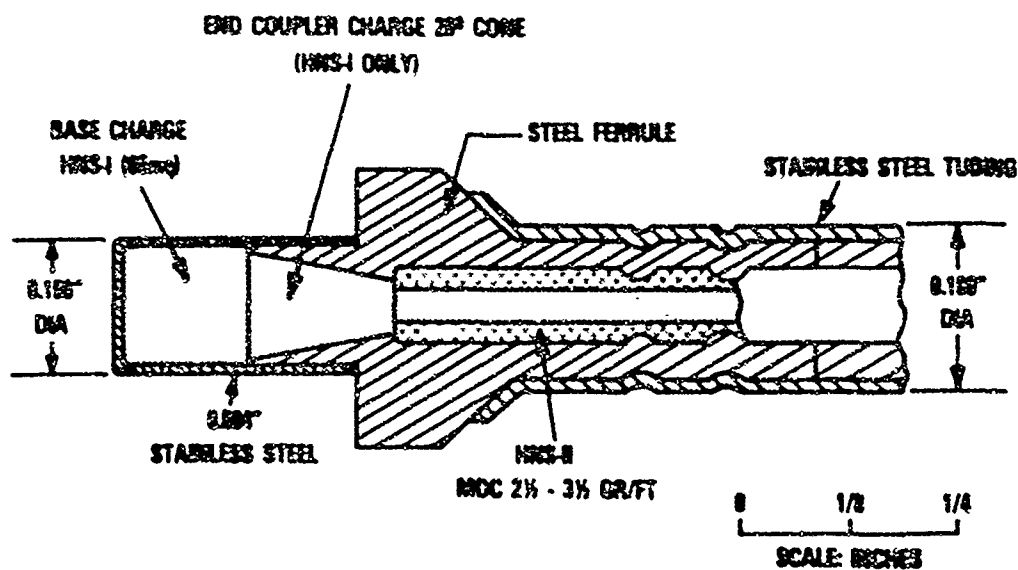


FIGURE 2 SMOG END BOOSTER



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FIGURE 3 CRYSTAL GROWTH HNS-1

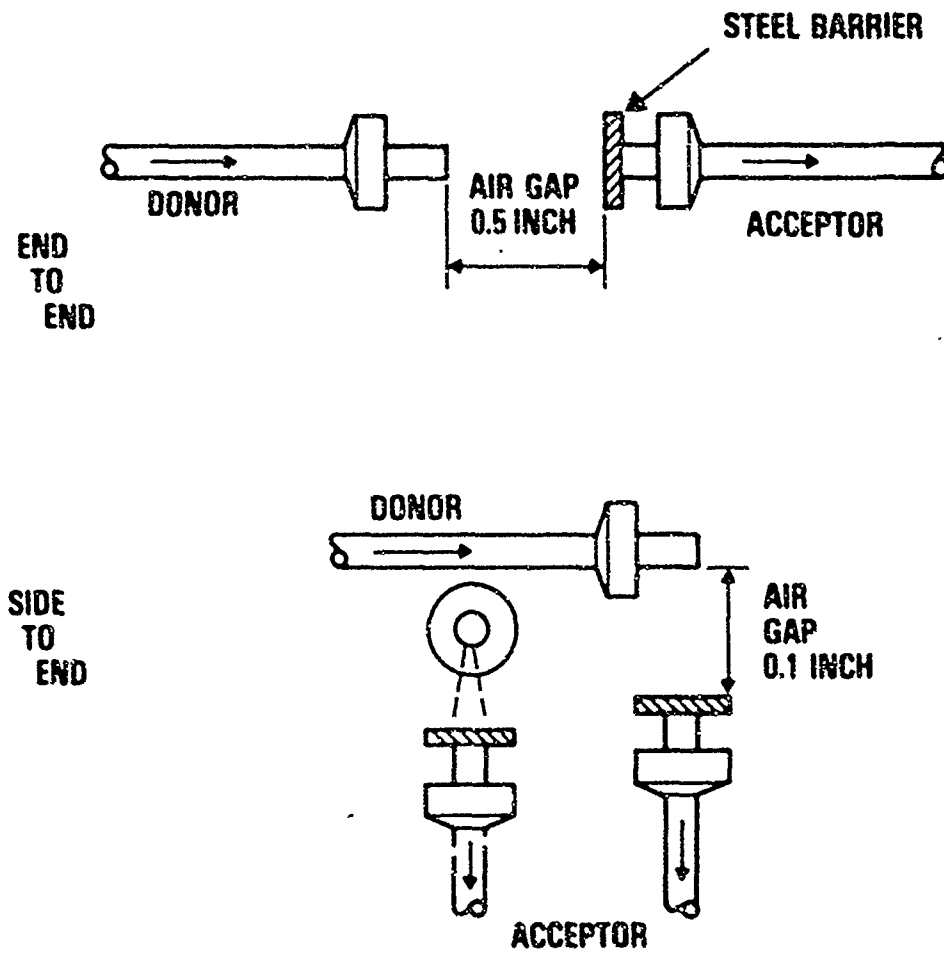


FIGURE 4 DETONATION TRANSFER ARRANGEMENT McDONNELL DOUGLAS COOPERATION

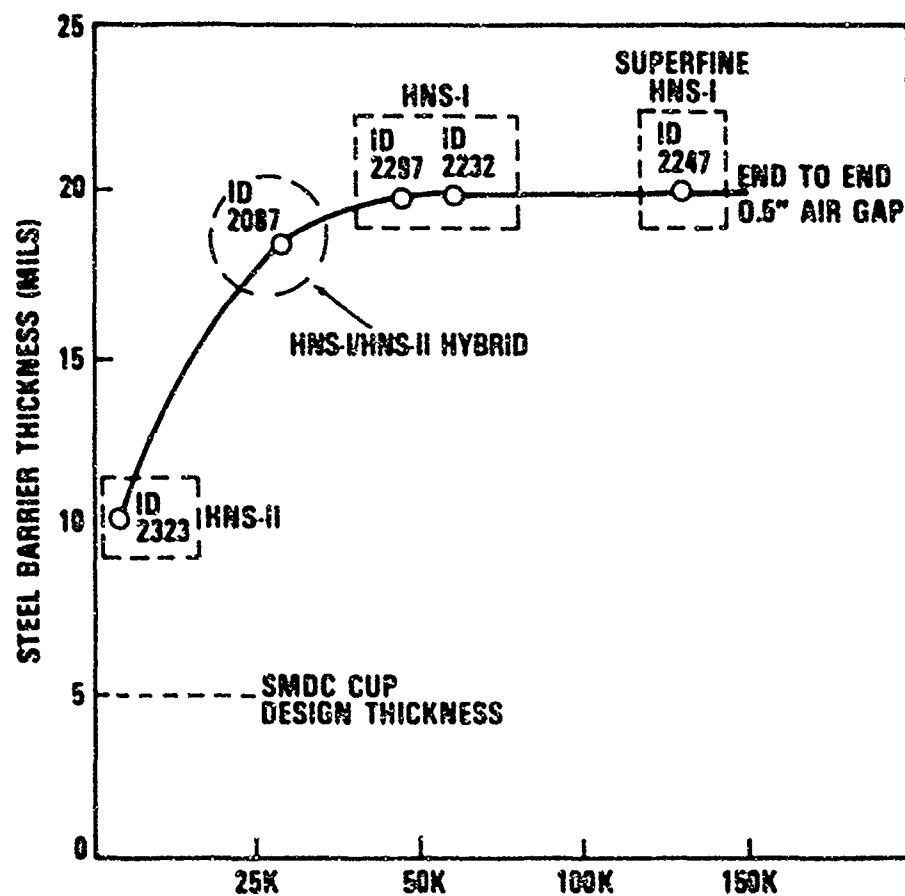


FIGURE 5 HEXANITROSTILBENE-FRAGMENT INITIATION SENSITIVITY AT A 50% FIRE RESPONSE WITH STEEL BARRIER THICKNESS VS EXPLOSIVE SURFACE AREA AT THE 0.5" AIR GAP

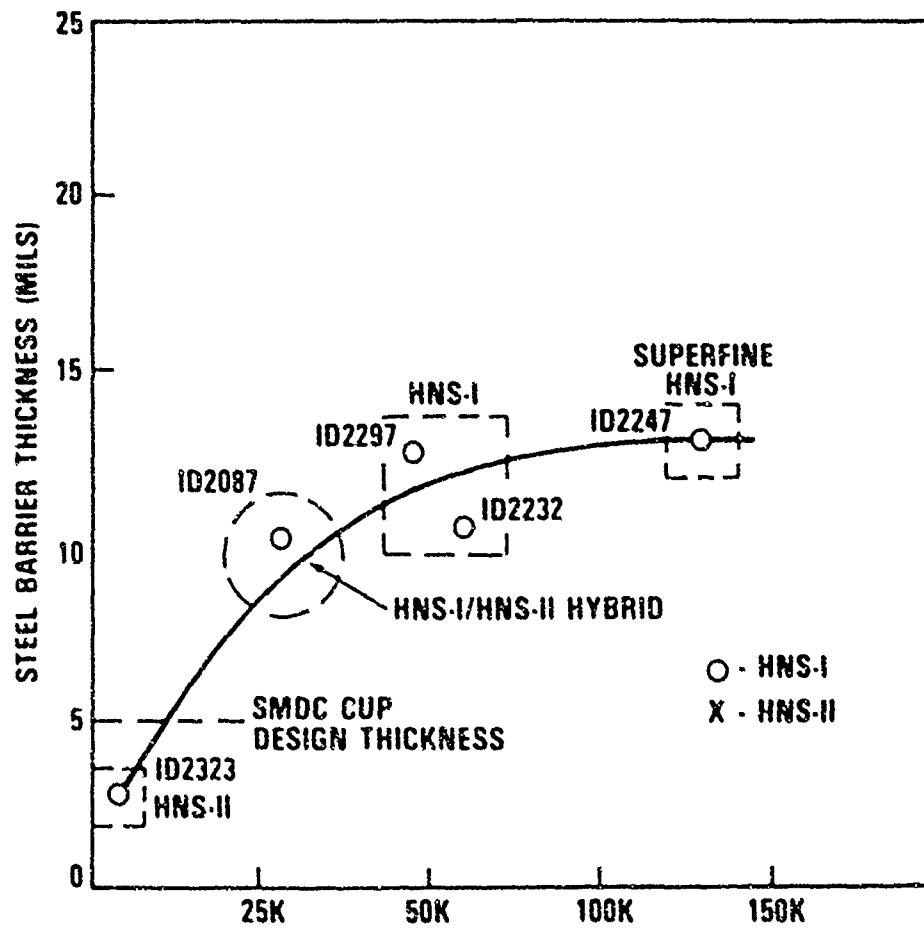


FIGURE 6 HEXANITROSTILBENE-FRAGMENT INITIATION SENSITIVITY AT A 60% FIRE RESPONSE WITH STEEL BARRIER THICKNESS VS EXPLOSIVE SURFACE AREA AT THE 0.1" 100 AIR GAP

TABLE 1 QUANTITATIVE HNS DATA

% COMPOSITION DETERMINED FROM RESPONSE FACTORS

SOURCE	% HNS-I	% HNS-B	% TNB	% PiCH ₂ OH	% PiCHO	% TNT	% "P.P."
A	99.4+	0.4	0.2				
B	93.9+	1.6	0.2			0.1	0.8
C	95.9+	0.6-4	TRACE			0.5	0.1
D	99.0+	0.7					0.2
E	98.0+	1.7	0.2	TRACE	TRACE		
A	HNS-II 99.4	0.5					
B	99.6+	0.1					0.3
C	98.2+	0.3				0-0.1	0.3
D	99.1+	0.7					0.2
E	99.0+	0.5					0.5

TABLE 2 AVAILABILITY-HEXANITROSTILBENE (HNS)

SOURCE	TYPE
CHEMTRONICS	HNS-I HNS-II
MASON & HANGER- SILAS MASON CO., INC. PANTEX PLANT	HNS-I HNS-II
TELEDYNE McCORMICK SELPH	HNS-I HNS-II
UNITED TECHNOLOGIES CORP	HNS-I
ENSIGN BICKFORD	HNS-II
BRITISH (ROF-BRIDGEWATER)	HNS-I

TABLE 3 DETONATION VELOCITY/CHEMICAL ANALYSIS AFTER ELEVATED TEMPERATURE STORAGE @ 425°F HNS-II SILVER MDC.

TESTING YEAR	EXPOSURE TIME (HOURS)	t ₀	t ₂	t ₄	t ₈	t ₂₀	t ₂₈	t ₃₂	t ₄₈	t ₅₆
1974	DETONATION VELOCITY (m/s)	7130	-	6631	6250	-	-	5541	-	(b)
1978	DETONATION VELOCITY (m/s)	6750	6195	6161	(a) EXPL COLUMN EXTRUDING FROM MDC SHEATH	(b)	(b)			
CHEMICAL ANALYSIS 1978	REMAINING % HNS	100.00	-	100.00	81.3	17.6	3.4	-	-	-
	% SOL MAT	0.00	-	0.00	11.1	59.0	27.8	-	-	-
	% INSOL MAT	0.01	-	0.01	5.8	23.4	68.8	-	-	-

*ID 1850 HNS-II 2 1/2 GR/FT SILVER SHEATH VENDOR : EXPLOSIVE TECH (NO ANNEALING OF CORDS)
CONTAINS HNS-II ID 1859 (T/MC/S LOT 8203.2 NITRIC ACID RECRY'S HNS). NITRIC ACID ASSAY % 0.05

(a) IN LINE FAILURE: COLOR CHANGE IN SOLUTION DMSO
(b) INITIATION FAILURE

TABLE 4 EXPLOSIVE TECHNOLOGY SMDC SUMMARY (MAJOR PROGRAMS ONLY)

1965 THROUGH JANUARY 1978

PROGRAM	QUANTITY MANUFACTURED	QUANTITY FUNCTIONED	X-CORD CONFG.	
			CORE 2.5 GR/FY	SHEATH MAT'L
F-111	281,050	17,730	DIPAM	SILVER
F-14	123,940	11,550	HNS	SILVER
F-15	15,870	980	DIPAM	SILVER
F-16	230	40	HNS	SILVER
S-3A	16,530	830	HNS	SILVER
EA-6B	3,500	400	HNS	SILVER
MB-339 (ITALY)	90	10	HNS	SILVER
C-101 (SPAIN)	140	20	HNS	SILVER
SPACE SHUTTLE	1,750	200	HNS	SILVER
DELTA CASTOR	1,870	180	HNS	SILVER
PROJECT 227	3,230	250	DIPAM	SILVER
LM CUTTER	2,300	1,050	HNS	SILVER
CENTAUR	400	80	HNS	SILVER
CTS (CANADA)	80	30	DIPAM	SILVER
STANDARD ARM	4,500	120	DIPAM	SILVER
SPRINT	1,000	100	RDX	LEAD
HARPOON	710	70	HNS	SILVER

TABLE 5 PHYSICAL AND EXPLOSIVE PROPERTIES OF HEXANITROSTILBENE,
HNS/TEFLON COMPARED TO RDX

	HNS-I	HNS-II	HNS-II/TEFLON 90/10	STD RDX***
MELTING POINT (°C)	313	318	318	204
THEORETICAL MAXIMUM DENSITY (G/CC)	1.74	1.74	1.78	1.82
VACUUM THERMAL STABILITY 260°C (CC/G/HR)	1.68	0.23	250° 0.52	13.2 AT 180
WEIGHT LOSS (%) AT 210°C AFTER 48 HRS	—	—	—	EXPLODES
PARTICLE SIZE RANGE (MICRONS)	< 10	100-200	100-200	600 OR LESS
IMPACT SENSITIVITY (X IN CM)*	47	63	—	20
ELECTROSTATIC SPARK SENSITIVITY	FIRES ABOVE 0.001 MFD Ø 8 KV	FIRES ABOVE 0.0001 MFD Ø 17 KV	—	LESS SENS THAN TETRYL OR PETN
DETONATION VELOCITY (M/SEC @ DENSITY (G/CC))	6800 1.60	7000 1.70	6900 1.68	8350 1.70
50% SHOCK SENSITIVITY DB _g ** (KBAR) Ø DENSITY, G/CC	7.14 (33.89) 1.68	5.35 (18.74) 1.64	5.76 (21.87) 1.70	3.77 (11.26) 1.63
AVAILABILITY	PRODUCTION	PRODUCTION	—	PRODUCTION
SPECIFICATION	WS5003	WS5003	NOLS 1015	MIL-R-398
*NOL-ERL DROP MACHINE, SANDPAPER, TYPE 12 TOOLS, 2.5 KG WT **DB _g = 30-10 LOG (OBSERVED GAP IN MILS) ***MIL-R-398 MILITARY SPECIFICATION RDX				

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To all holders of NSWC TR 79-181
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Change 2
31 March 1980

1 page

This publication is changed as follows:

TABLE 5, page 21/22, PHYSICAL AND EXPLOSIVE PROPERTIES OF HEXANITROSTILBENE, HNS/TEFLON COMPARED TO RDX.

The THEORETICAL MAXIMUM DENSITY (G/CC) for STD RDX reads 1.82. This value should be changed to read 1.806.*

The footnote should read:

*UCRL-51319 (Revision 1976), Brigitta Dobratz, Lawrence Livermore Laboratory.

Insert this change sheet between the cover and the DD Form 1473 in your copy.
Write on the cover "Change 2 inserted"

[Signature]
ELIHU ZIMET, Acting Head
By direction